

BIOGAS PRODUCTION BY TWO-STAGE THERMOPHILIC AND MESOPHILIC BIODIGESTION OF KITCHEN WASTE

A Thesis submitted in partial fulfillment of the requirements for the degree of

***Bachelor of Technology
In
Biotechnology***

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CERTIFICATE OF APPROVAL

This is to certify that the thesis entitled “**BIOGAS PRODUCTION BY TWO-STAGE THERMOPHILIC AND MESOPHILIC BIODIGESTION FROM KITCHEN WASTE**” submitted by **Rahul Kumar** has been carried out under my supervision in partial fulfillment of the requirements for the Degree of *Bachelor of Technology* in *Biotechnology Engineering* at National Institute of Technology Rourkela and this work has not been submitted elsewhere for any other academic degree/diploma to the best of my knowledge.

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ABSTRACT

The present research work focuses on the biogas production from kitchen waste generated at the NIT hostels and to investigate the effects of the key process parameters like pH and temperature, by operating a pilot scale setup in two stage thermophilic and mesophilic digestion conditions. In 1st experimental setup, a comparison of the digestion of food wastes in small scale was done. In this experiment three setups were operated in mesophilic, thermophilic and two stage mesophilic and thermophilic conditions respectively. The comparison of kitchen waste digestion in case of two stage setup was found to be 30% faster than mesophilic setup in terms of total solids and carbohydrates degradation over the operation period of 20 days. The biogas production initially was found to be 40% faster in case of two stage setup than the mesophilic setup.

In 2nd experimental setup the pilot scale setup was based on two stage thermophilic and mesophilic digestion process and operated as a batch reactor. In batch condition, maximum of 7.45 Liters biogas was produced from the digestion of 6 kg of food wastes in 25 days. The initial total solid content of the waste slurry was measured to be 10.27% which was reduced to 5.51% on 25th day. The initial total carbohydrate and volatile fatty acid concentration was 61.2 g/L and 2475.5 mg/L respectively. After 24 days of digestion, the total carbohydrate concentration was decreased to 22.3 g/L, whereas volatile fatty acid concentration was increased to 4954 mg/L. The project work signifies that the kitchen waste can be used as a potential source for biogas production using two stage digestion process and thus effective waste management can be achieved.

CHAPTER 1

INTRODUCTION

Anaerobic digestion is one of the most widely used, proven processes that is being used for the treatment of the solid wastes. Anaerobic digestion processes has its history dating back to the 18th century. Since the mid-19th century the role of anaerobic bacteria has been understood in the digestion process. It has been over a century, since anaerobic digestion has been used for the treatment of sewage and cattle dung slurry. The history of anaerobic digestion technology in India dates back to late 19th century when the first biogas plant was established in Matunga (Mumbai) in the year 1897 [1].

Anaerobic digestion is a process in which the biological processes like biodigestion by the microbes occur. Anaerobic digestion processes breakdown the organic matter in the feed materials in anaerobic conditions i.e., in the absence of oxygen. These processes stabilizes these waste materials against rapid decomposition. The conversion process is conservative in nature which produces a stable digestate that can be used as a bio-fertilizer. The methane gas and carbon dioxide are also produced which are together known as biogas [1]. Thus in addition to treatment of the solid wastes, anaerobic digestion also allows recovery of energy value by conversion of the volatile solids into biogas. The process also functions as a waste material disposal system.

The Biogas produced by anaerobic digestion process has methane as its major constituent. Biogas is a renewable energy source that is used as a fuel [1]. This Biogas can be used as a fuel to produce heat, through combustion. Biogas is also used at many places across the world for production of electricity in combined heat and power (CHP) system. The CHP systems in addition to meeting the energy required for the functioning of the biogas plants also produce enough energy that can be further used to produce electricity.

Kitchen wastes consists of uncooked and cooked solid food wastes discarded from the kitchens of houses, restaurants, hotels, messes, etc. These food wastes have high organic content with high nutritive value for the microbes, which can utilize the organic materials as nutrients and in return reduce the wastes to biogas and digestate. These wastes usually end up landfills or dumped in some open land where they degrade in the open. The insects and animals feed on these wastes, and sometimes pathogenic microbes also grow on these discarded food wastes. These pathogenic microbes spread by vectors like flies, mosquitoes, rats and other disease bearing vectors and are the cause of public health hazards and various types of diseases in humans like cholera, diarrhea, typhoid, etc.

If these food wastes are allowed to degrade in a controlled environment by anaerobic digestion in specifically designed digesters then the problem of dumping these wastes can be solved and by use of several methods higher efficiency of methane production obtained, which reduces the cost of production of biogas. Thus we are able to extract energy value from the wastes materials and even reduce the adverse effects from dumping of the wastes materials.

Various researches have been conducted to improve the production and yield of the biogas. Earlier the biogas plants in India were operated with animal dung as slurry and the gas produced was also known as gobar gas. But with time the type of substrate used in the biogas production has changed. In some parts around the world huge biogas plants have been developed which operate with agricultural wastes and food wastes as substrates. Various technological innovations and alteration of the working conditions of the biogas plants has resulted in yield of biogas that has been much higher than it was in the conventional biogas plants.

Food wastes have become a major source of substrate for the biogas plants due to their high organic content. Food wastes has also been used as substrate in combination with animal dung in biogas plants to obtain an overall high production of biogas [2].

Two stage biogas digester has been under research for the treatment of kitchen wastes. The two stage setup has showed to decrease the retention time of the digestion process considerably. In conventional mesophilic biogas plants the retention time is 30 to 50 days on an average. The retention time in mesophilic biogas plants has been found to decrease below 15 days in large scale implementation [3]. A large scale biogas plant based on food wastes can be implemented at NIT Rourkela campus considering the huge amount of food wastes generated from the hostels messes at the campus.

CHAPTER 2

LITERATURE REVIEWS

2.1 BIOGAS CHARACTERSTICS

Biogas has methane as its main constituent that is that is produced by the anaerobic biodegradation of the organic material of the wastes by microorganisms in anaerobic conditions. It results in residual waste which is of superior nutrient quality as a fertilizer.

The usual composition of biogas is [4]:

- Methane (50% - 70%)
- Carbon dioxide (30% - 40%)
- Hydrogen (5% - 10%)
- Nitrogen (1% - 2%)
- Water vapor (0.3%)
- Hydrogen sulfide (traces)

The Biogas produced may vary in composition depending on the feed material. Biogas is lighter than air by 20% and the ignition temperature of biogas lies in the range 650 °C to 750 °C. Biogas is a colorless gas which burns with blue flame. The biogas can be used as a fuel in substitution to firewood, LPG, etc. and can also be used to produce electricity. Biogas has a calorific value of about 20 Mega Joules (MJ) /m³ and has been reported to burn with 60 % efficiency when used for combustion in a biogas stove. Biogas has been found to have energy content of 6-6.5 kWh/m³. Biogas is equivalent to 0.6-0.65 l oil/m³ of biogas and it may explode when present in air at concentration of 6-12 % of air. Biogas has critical temperature of -82.5 °C, density of 1.2 kg/m³ and usually smells like bad eggs [4-6].

The residual organic matter that is obtained after digestion of the feed material is rich in nutrients, like phosphates and can be used as a bio fertilizer. Anaerobic digestion of the human wastes not only serves as an energy retrieval system but also acts as a valuable waste disposal system in case of wastes like human wastes, kitchen wastes, agricultural wastes, etc. reducing the problem of dumping these wastes and the contamination due to these waste materials.

2.2 BIOGAS PRODUCTION PROCESS

The general Biogas production system consists of the following stages [4, 6]:

- **Waste collection:** The waste materials from various sources are collected and segregated. The materials like plastics that cannot be digested by the microbes are removed before the wastes are added to the digester so that they do not affect the activity of digester.
- **Pre-treatment:** In this stage the waste materials are treated with water or other chemicals which aid in the digestion of these wastes.
- **Homogenization:** In this stage the wastes are mixed and crushed in homogenizers to breakdown large particles into smaller ones as the smaller particles are easily digestible by the microbes.
- **Feeding:** The substrate materials are fed to the digester tanks where water and other materials are added to allow the digestion of the wastes.
- **Anaerobic Digestion:** The wastes are digested by the various microbes involved in the process. The maintenance of pH, temperature and other factors influencing the digestion of the wastes for optimum digestion of the substrate and the production of biogas.
- **Production and utilization:** The biogas produced due to the anaerobic digestion of the wastes concentrated in this stage by cleaning and removing contaminant gases. This biogas can be directly used by combustion. The sludge that is produced as by product is dried to remove water. This sludge can be utilized as fertilizer as it is rich in nutrients like phosphates, nitrates.

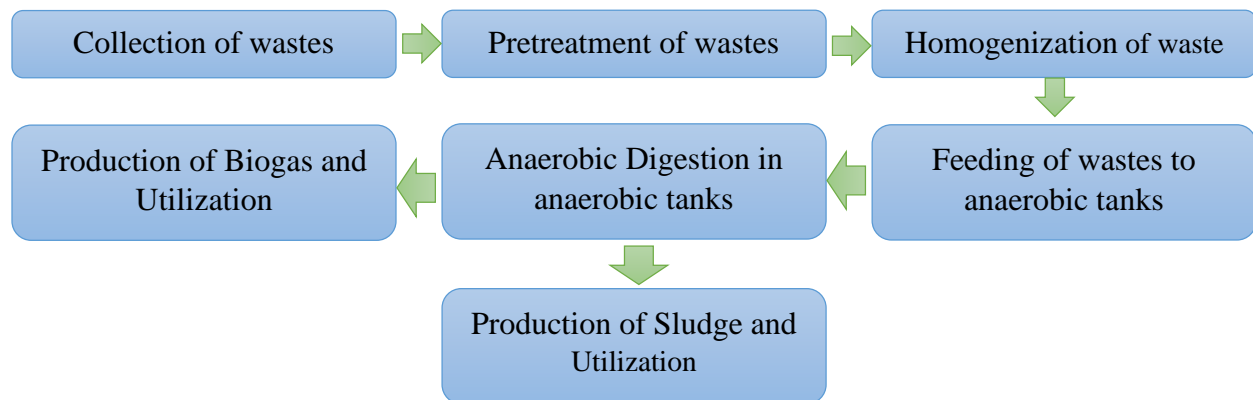


Figure 1: Schematic Diagram of Biogas Production Process

2.3 PRINCIPLE OF ANAEROBIC DIGESTION

Anaerobic digestion is a process which occurs in absence of oxygen. During this process various microbes are involved which breakdown the organic substances through various biochemical processes that finally result in biogas and digested sludge that is rich in nutrients. Overall anaerobic digestion process is a symbiotic process in which different bacteria involved depend upon each other.

The anaerobic digestion process consists of three stages [2, 3]:

- **HYDROLYSIS:** It is the first step in the anaerobic digestion process. The waste materials produced from plant and animal origin consists mainly of carbohydrates, lipids, proteins and other inorganic materials. During hydrolysis large molecular complex substances are broken down into simpler molecules like glucose by bacteria involved in the process with the help of enzymes such as celluloses, proteases, amylases and lipases released by those bacteria. The important bacteria involved at this stage are (i) Clostridium, (ii) Vibrio, (iii) Bacillus, (iv) Micrococcus and (v) Peptococcus. This stage is also popularly known as the polymer breakdown stage [4].
- **ACIDIFICATION:** It is the second step of the process. In this step the glucose that is produced during hydrolysis is utilized by acid producing bacteria during this stage. During the process the bacteria convert these molecules into various acids like acetic acid, butyric acid, propionic acids and ethanol. During this process hydrogen and carbon dioxide are also produced. The acid producing bacteria during this process also consume all the oxygen and helps in creating conditions suitable anaerobic conditions for the growth of methanogenic bacteria. The important bacteria involved in this stage of the process are (i) Clostridium (ii) Rumino coccus, (iii) Propioni bacterium and (iv) Desulphobacter streptococcus.
- **METHANOGENESIS:** This stage is the last stage of the anaerobic digestion process and the rate determining step of the process. In this stage the methane producing bacteria are involved. These bacteria utilize the low molecular weight compounds like the acetic acid produced during the previous steps as nutrients for themselves and in the

process form methane and carbon dioxide gas which together constitute the major part of the biogas. The important bacteria involves methanogenic stage are (i) Non- sporulating methanobacterium, (ii) Sporulating methanobacterium and (iii) Sarcinaea.

The whole biochemical process is summarized by the “**Buswell**” formula [1]. This formula gives the total stoichiometric relation of the complete anaerobic digestion process:

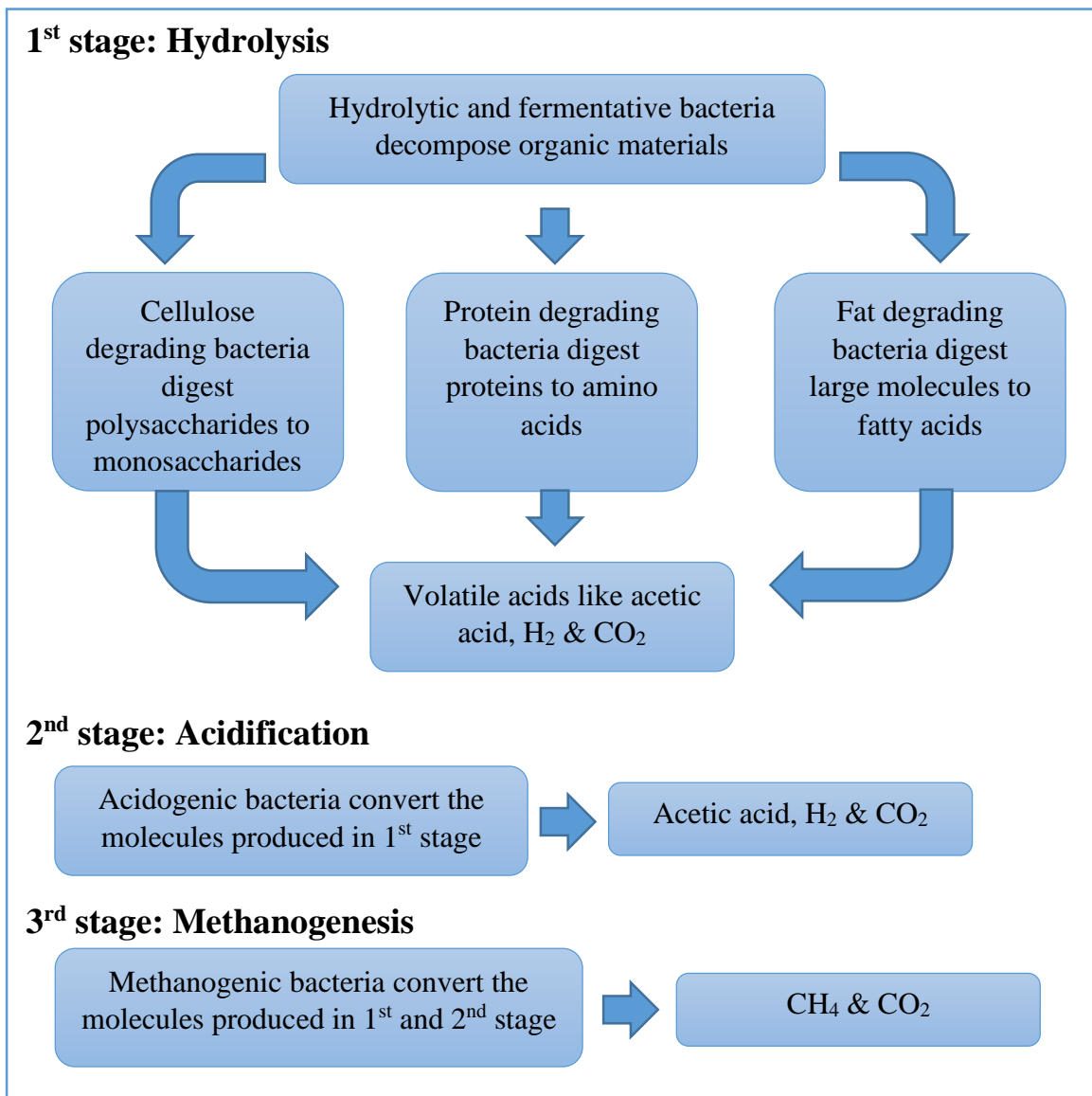
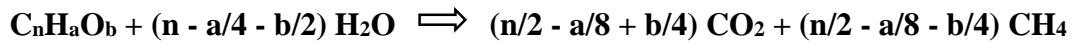


Figure 2: Schematic Diagram of Mechanism of Anaerobic Digestion

2.3.1 THERMOPHILIC AND MESOPHILIC DIGESTION

Thermophilic Digestion: It is the process which involves thermophilic bacteria and the digestion process takes place at temperatures above 50 °C. The process is advantageous because little or no agitation of the substrate may be required during the process and the digestion process may be almost ten times faster than in case of mesophilic digestion. This type of digestion kills the pathogenic bacteria that may develop during the process due to high temperature of operation. The hydraulic retention time of the substrate in the digesters is low as the digestion is faster. The destruction of volatile solids is faster thus it can also work at higher loading rate. The disadvantages of this process is that energy is required to heat the source substrate and the process may not be as stable as mesophilic process [7-12].

Mesophilic Digestion: It is the process which involves mesophilic bacteria and the digestion process takes place at temperature range between 29 °C to 40 °C. This process is the conventional process that is usually used for anaerobic digestion in digesters. The advantage of this process is that it is stable and the gas production obtained may be at almost constant rate if operated at optimum conditions of pH and temperature. No external energy is required for the operation of this process as the heat produced during the process is enough to maintain the temperature conditions of the process. The disadvantage of this process is that it is very slow. The retention time of the substrate materials required is very high and may require even up to 45 to 60 days for complete digestion of the substrate [7-12].

The conventional biogas production process is based on mesophilic digestion of the substrate but with technological advancement thermophilic digestion is also researched in various cases ^[7-12]. Various researches have also been conducted to compare the two of the above processes for their feasibility and determination of functional parameters [13].

2.3.2 TWO STAGE ANAEROBIC DIGESTION

The two stage digestion of the substrate material is also a gaining area of interest. The substrate in this type of digestion is digested in two stages in which the substrate is subjected to different conditions in each stage [3, 14]. The first stage is usually the thermophilic stage as the hydrolysis of the substrate is faster at thermophilic temperatures. The acidification also occurs at a faster rate thus creating conditions suitable for the methanogenic bacteria to grow in next stage. The faster degradation of the substrate reduces the retention time of the substrate in this stage which may be as low as 2 days [14].

The substrate is then subjected to mesophilic conditions in second stage where the substrate is digested at mesophilic temperatures [3, 14-18]. The substrate when in mesophilic stage the methanogenic bacteria develop as the optimal temperature for the activity of most of the methanogenic bacteria is in the mesophilic range. These bacteria are able to grow rapidly in the anaerobic conditions created during the first stage and thus biogas production is faster as compared to conventional only mesophilic process. As the development of bacteria is faster the retention time in this case is also very much reduced and may be as low as 5 days.

The advantage of the two stage process is that the retention time required for the digestion of the substrate is very low and it has been reported that the total digestion time of the waste may be less than 15 days [3, 14-18]. As the digestion time is low more amount of waste can be processed on a daily basis thus the size of the digester required is smaller than mesophilic digesters. The two stage process also overcomes the problems of instability in case of thermophilic digesters and stable gas production is obtained. The quality of biogas obtained in the two stage process is also high and the methane content of the gas may be as high as 70-75% as compared to 50-55% in case of conventional mesophilic digester [3, 14].

2.4 FACTORS INFLUENCING PRODUCTION AND YIELD OF BIOGAS

The various factors which effect the biogas production in a digester are [4, 19]:

- **C/N Ratio:** Carbon to Nitrogen ratio has a huge influence on the anaerobic digestion process. If C/N ratio in the digester substrate is very high, Nitrogen present in the substrate will be consumed too rapidly as compared to carbon, by the methanogenic bacteria to meet their protein requirements and thus the function of the bacteria will be effected as they will no longer be able to act on the remaining carbon content, resulting in low gas production. If the C/N ratio in the digester is very low then Nitrogen may get liberated as ammonia and thus increasing the pH value of the digester contents. Thus ammonia accumulation can lead to low gas production as the pH greater than 8.5 in the digester becomes toxic for the operation of the methanogenic bacteria. The optimum C/N ratio is 20:1 to 30:1 for anaerobic digestion of the substrate material.
- **Dilution and Loading rate:** The dilution of the substrate feed material is important for the functioning of the anaerobic digester. If the substrate material is too dilute then the solid content of the substrate tends to settle down into the digester. If the substrate material is too thick, the flow of the gas produced is hampered. In both the cases the gas production obtained is less than the optimum. The optimum dilution rate should be 7% to 10% of the total solids in the substrate material.
- **pH value:** The pH of the substrate feed material has the greatest influence on the biogas production. Different bacteria involved in the anaerobic digestion process have different optimal pH ranges thus their activity is effected with change in pH of the substrate. The pH of the digester can decrease below 5.0 due to large amount of organic acids that are produced during initial stages. This pH value is not favorable for the growth of the methanogenic bacteria. Thus pH of the digester needs to be maintained around 6.5 during the acidification step. The pH during the methanogenic step may go up to 8.5 or more due to higher ammonia production but it is not favorable thus pH needs to be maintained around 7.0 for the optimal functioning of the methanogenic bacteria [3].

➤ **Temperature:** The temperature at which the digester is operated influences the biogas production. The digester can be operated at three temperature ranges (i) Psychrophilic temperature range (below 35 °C), (ii) Mesophilic temperature range (between 29 °C to 40 °C) and (iii) Thermophilic temperature range (between 50 °C to 55 °C). The biogas production differs greatly due to change in temperature. In case of thermophilic temperature operation of reactor biogas production might be higher and the retention time of the substrate is low due to faster digestion by thermophilic bacteria, but the methane content of the biogas may be low because the optimum temperature of the methanogenic bacteria lies in mesophilic range. In case of mesophilic temperature operation of the digester the process is slow and the retention time of the substrate material is also high. But the biogas production is stable when the digester is operated at optimum conditions [3-7].

➤ **Hydraulic Retention Time (HRT):** HRT is the average time period for which the given quantity of input substrate remains inside the digester for digestion. The effective retention time of digestion ranges from 30 to 55 days, depending on the type of substrate used and the optimum pH and temperature at which the digester is operated [3]. The addition of other substances which aid the digestion process and help in the faster digestion of the substrate also influence the retention time. Various researches have shown that the retention time of the digester can be reduced even to as low as 15 days.

➤ **Toxicity:** The micronutrients like copper, nickel, zinc, etc. are essential for the growth of the bacteria. But presence of high concentration of these nutrients can cause toxicity to the microbes in the digester. The concentrations of these nutrients must be below 1.5mg/L. The micronutrient cobalt in its optimum concentration increases the methane production.

2.5 BENEFITS OF THE BIOGAS TECHNOLOGY

- The economic benefits obtained are:
 1. The waste materials are treated at without becoming a problem to the environment in the form of air and water pollution.
 2. Fertilizer is obtained as a byproduct of the process which are rich in nutrients and can be directly used as manure in farms to improve soil fertility.
 3. Biogas produced is a renewable energy source which can be used directly in cooking stones as an alternative of LPG or can be used to produce electricity.
 4. The landfill space required for the dumping of the waste materials is reduced thus reducing the land requirement.
- The social and health benefits obtained are:
 1. Biogas plant operation requires manpower for its operation thus creating job opportunities.
 2. The fertilizer obtained are cheap and thus can be readily available to the farmers at low cost.
 3. The chances of spreading of diseases causing microbes is reduced.
 4. Environmental hazards are prevented through reduction of soil, water and air pollution.

CHAPTER 3

OBJECTIVES

- [1] To produce Biogas using only kitchen waste as substrate.
- [2] To analyze of the characteristics of the food wastes generated from NIT campus hostels.
- [3] To compare the gas production in two stage thermophilic and mesophilic digestion of kitchen wastes with the mesophilic and thermophilic digestions.
- [4] To understand the effects of various process parameters on gas production.
- [5] Determination of the amount of wastes generated from the NIT hostels.
- [6] Optimization of the biogas production parameters in two stage process thermophilic and mesophilic digestion of kitchen wastes.
- [7] To check the optimization of the gas production parameters by making the working model of a pilot scale plant.

CHAPTER 4

MATERIALS AND METHODS

4.1 EXPERIMENTAL PROCEDURES

4.1.1 EXPERIMENT: 1. TOTAL & VOLATILE SOLIDS

These tests have been performed to estimate the amount of organic substance present in the waste stream. The total solids percentage gives an estimate of total solid content present in the sample in bio-digester setup. The volatile solids percentage gives a rough estimate of the organic content because the loss of solid content after ignition might also be due to degradation and volatilization of minerals instead of organic matter. Therefore, a confirmatory test was also required. Following tests have been performed as per standard methods with slight modifications [20].

TOTAL SOLIDS (TS %)

Total Solids (TS %) is the amount of solid present in the sample after the water present in it is evaporated.

FIXED SOLIDS and VOLATILE SOLIDS (VS %)

Fixed Solids (%) is the amount of solids left behind after the total solids obtained is dried at very high temperature of 550 °C.

Volatile Solids (VS %) is the amount of solids that evaporated after the total solids obtained was dried at very high temperature of 550 °C.

Materials used:

- Crucibles
- Hot air oven
- Muffle furnace
- Weighing balance
- Food waste sample

Procedure:

- The initial weight of the dry empty crucible was measured.
- A gm of food waste sample was taken into dry crucibles from the digester.
- The crucibles were heated in oven at 103 °C-105 °C for 2 hours.
- The crucibles containing samples were then cooled in desiccator.
- The crucible was weighed and the weight was noted down. Then the crucible was again heated in the oven as mentioned above.
- This cycle of drying, cooling and weighing was repeated until a constant weight was obtained (B gm).
- The total solids obtained in the crucible was ignited for 15 minutes at 550 °C.
- The crucible was cooled in a desiccator and then weighed to obtain final weight of the solids left behind in the crucible (C gm).

Calculations:

$$\begin{aligned} \text{Total solids as \% sample} &= (\text{Final weight} / \text{Initial weight}) * 100 \\ &= (B/A) * 100 \end{aligned}$$

$$\text{Volatile solids as \% total solids} = [(B - C)/B] * 100$$

$$\text{Volatile solids as \% sample} = [(A - C)/A] * 100$$

Where, A = initial wt. of wet samples, in gm.

B = final wt. of dried residue after heating at 100-105 °C, in gm.

C = final wt. of dried residue after heating at 550 °C, in gm.

4.1.2 EXPERIMENT: 2. TOTAL CARBOHYDRATES

Phenol - Sulfuric Acid Method was used to obtain the quantitative amount of carbohydrates in the digester samples [16].

In hot acidic medium, glucose is dehydrated to hydroxymethyl furfural. This forms a yellow-brown colored product with phenol and has absorption maximum at 490 nm.

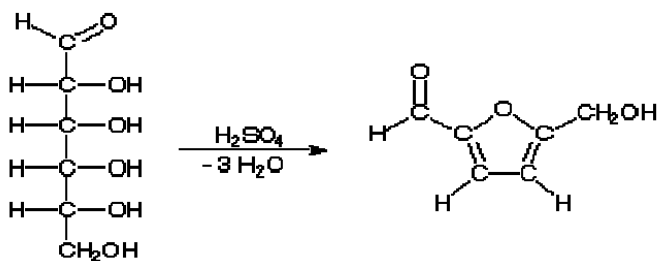


Figure 3: Chemical Reaction for Phenol Sulfuric acid

The Procedure used has been taken from the Himedia Carbohydrates Estimation Teaching Kit with slight modification.

Procedure:

1. Standard Glucose stock solution was prepared with concentration of 1mg/ml.
2. Eight clean and dried test tubes were and labelled as Blank and 1-7.
3. Dilutions of glucose standards were made with concentrations of 20, 40, 60, 80 and 100 mg per 1ml by transferring respective amount of glucose from the standard glucose solution (1mg/ml) and adjusting it to a total volume of 1ml by adding distilled water as mentioned in Table 0.
4. The sample used were taken from the digester and diluted 10 times and then 1ml of the sample solution was taken in tubes 6 and 7.
5. Then 1 ml of 5 % phenol solution was added to all the tubes.
6. Then we added 5 ml concentrated Sulfuric acid to each tube.
7. After 10 minutes, the contents of the tubes were mixed and placed in a water bath set at 25-30 °C for 20 minutes.
8. The absorbance of all the tubes (1 – 7) was measured at 490 nm in a spectrophotometer.
9. A standard curve of absorbance at 490 nm on “Y” axis versus concentration of glucose in mg/ml on “X” axis was plot.
10. The value of the concentration of glucose of the unknown sample was estimated from graph corresponding to the OD reading of the samples.

Table 0:

Tube No.	Blank	1	2	3	4	5	6	7
Conc. of Glucose (mg/ml)	0	0.2	0.4	0.6	0.8	1	Sample	Sample
Glucose stock soln. (ml)	0	0.2	0.4	0.6	0.8	1		
Water (ml)	1	0.8	0.6	0.4	0.2	0	1	1
5% Phenol solution (ml)	1	1	1	1	1	1	1	1
Sulfuric acid (ml)	5	5	5	5	5	5	5	5

Sugar concentration in the undiluted sample was calculated using the formula:

$$\begin{aligned}
 &\text{Concentration in undiluted Sample } \left(\frac{\text{mg}}{\text{ml}} \right) \\
 &= \text{Concentration of glucose in diluted sample } \left(\frac{\text{mg}}{\text{ml}} \right) \\
 &\quad * \text{Dilution factor}
 \end{aligned}$$

Dilution factor = 10

4.1.3 EXPERIMENT: 3. VOLATILE FATTY ACID (VFA)

VOLATILE FATTY ACID (VFA): - Volatile fatty acids (VFAs') are acids that are produced during the digestion of the organic wastes in anaerobic conditions during the acidification stage. These acids have carbon chain of six carbons or fewer. Examples of VFAs': acetic acid, propionic acid and butyric acid. Gas Chromatography is a method widely used to measure the quantitative and qualitative VFA analysis. Titration methods can be used for total VFA measurement. Two methods have been used during the experiment for total VFA measurement [6].

Method:

Titration procedure for measurement of VFA [6]:

1. 100 ml sample slurry was taken in a beaker.
2. The sample slurry was filtered using a filter paper.
3. pH of filtrate was checked and recorded.
4. 20 ml of filtrate was taken in a titration apparatus and 0.1M HCl was added to it until pH of the filtrate reaches 4. The amount of HCl added was recorded.
5. The filtrate sample was heated on the hot plate for 3 minutes.
6. The filtrate sample was cooled down and then titrated with 0.01M NaOH until the pH rose from 4 to 7. Amount of NaOH recorded.

$$\text{Total VFA content in } \frac{\text{mg}}{\text{l}} \text{ acetic acid} = (\text{Volume of NaOH titrated}) * 87.5$$

4.2 EXPERIMENTAL SETUPS

4.2.1 EXPERIMENTAL SETUP (Small Scale)

Three different digesters were setup under different conditions with same initial composition were installed as below [11]. The food waste used for the study in this project was collected from the mess of Vikram Sarabhai hall of residence at NIT campus, Rourkela. The food waste samples collected consisted of mixed wastes i.e., cooked wastes, uncooked wastes. The indigestible wastes like onion peels, small twigs, egg shells, etc. were removed from the collected food wastes manually.

The food wastes was then weighed 400gm for each setup. The food waste was crushed using a mixer grinder along with diluting it to 1 liter with water to form homogenized slurry(the food waste was diluted at a rate of 1:1.5 with water). The initial analysis of the food wastes before it was put into the reactor were performed, the same day it was collected from the hall. The analysis was done as follows.

- ❖ The diluted food wastes collected on the 1st day were analyzed for Total Solids (TS), Volatile Solids (VS), Total Carbohydrates (TC) and pH.

- ❖ The TS-VS content for each of the digester setup was measured every 5 days from the start of the setup.
- ❖ The total Carbohydrate content for each of the digester setup was measured using Phenol-Sulfuric acid method every 3 days from the start of the setup.
- ❖ pH was measured every 2 days and was adjusted by adding sodium hydroxide to regulate the pH around 7.
- ❖ Gas production of each setup was measured every day.
- ❖ The substrate in the setup was kept well mixed by shaking the setup 4-5 times a day.
- ❖ Hot water bath was used for heating and maintaining temperature during thermophilic stage.

Materials Used for the setup:

- 3 separate 1.2 liter plastic bottles.
- 400 gm grinded kitchen waste diluted to 1 liter with water for each setup.
- 100ml diluted cow dung slurry was added and used as inoculum.
- Rest space was left empty.

Setup: 1. 400gm food waste diluted with water to make 1litre then 100ml diluted cow dung slurry was kept under mesophilic conditions at room temperature. pH of the setup was measured and adjusted to 6.6 initially. **This setup was operated only for 20 days.**

Setup: 2. 400gm food waste diluted with water to make 1litre then 100ml diluted cow dung slurry. This setup was initially kept at thermophilic temperature (around 55° C) in a water bath for 2days and then under mesophilic conditions at room temperature and 50 ml more inoculum was added. pH of the setup was measured and adjusted to 6.7initially. **This setup was operated only for 20 days.**

Setup: 3. 400gm food waste diluted with water to make 1litre then 100ml diluted cow dung slurry was kept under thermophilic temperature (around 55° C) in a water bath. pH of the setup was measured and adjusted to 6.5 initially. **This setup was operated only for 10 days.**



Figure 4: Photographs of the small scale setups

4.2.2 EXPERIMENTAL SETUP (Pilot Scale)

The pilot scale digester was setup in Environmental Biotechnology lab of the Department of Biotechnology and Medical Engineering. The digester was setup based on the two phase design as showed below.

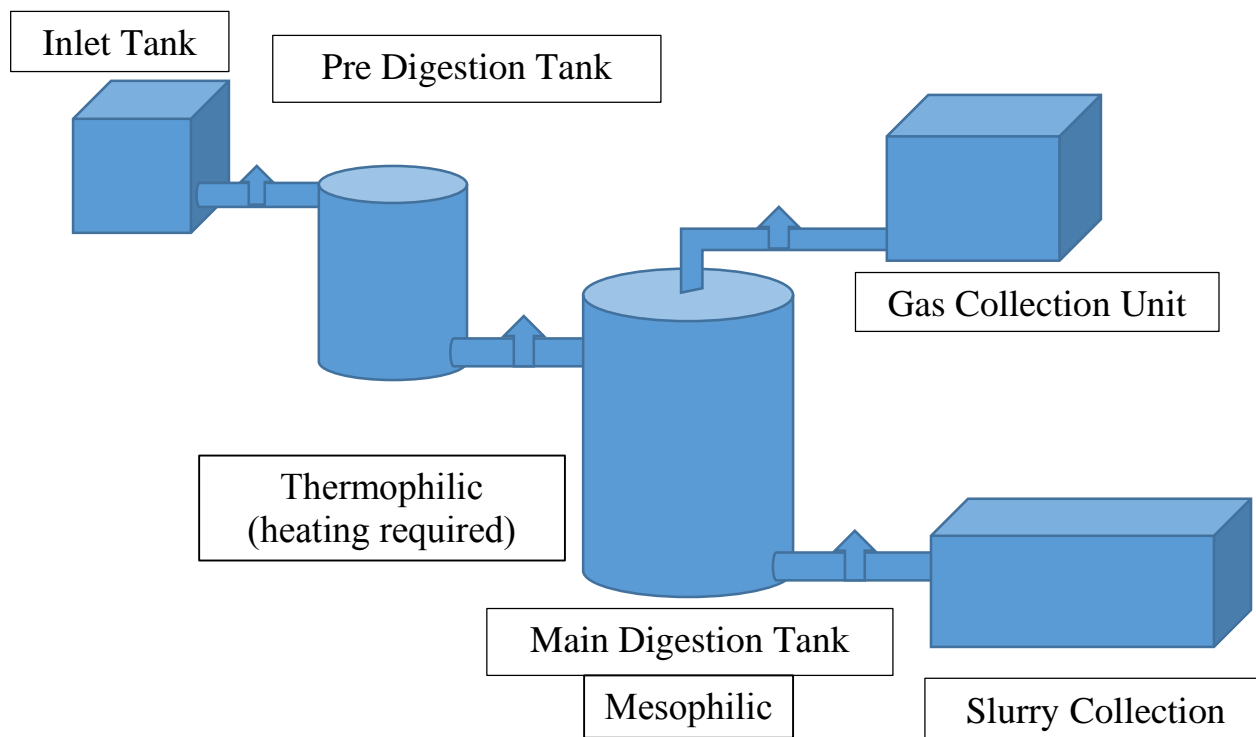


Figure 5: Schematic Diagram of the Pilot scale Biogas plant setup

The pilot scale biogas plant was made from the following components:-

1. **Inlet tank** - From this tank the crushed food waste material was to be added. This part was made from a plastic bucket in which the slurry was prepared then added to the pre-digestion tank through the inlet of that pre-digester tank.
Material used – Plastic Bucket (1 Nos.)
2. **Pre-digester tank** - here the waste materials were heated around temperature of 50-60°C so that thermophilic hydrolytic bacteria would grow. The substrate remained in this tank for 2 days. The heating helps in the faster digestion of the food waste.
Material used – Steel Cylindrical Tank (10 Liters capacity, 1 Nos.)
3. **Main digestion tank** – The waste materials after getting digested in the pre digester tanks for a 2 days was passed onto the main digester tank where the mesophilic methanogen bacteria was allowed to grow with the addition of some new inoculum, which digest the food wastes further to produce methane gas.
Material used – Steel Cylindrical Tank (20 Liters capacity, 1 Nos.)

- 4. Gas collection unit** – This unit was used to collect the gas that was generated from the digestion of the food wastes in the digester tanks. The gas was collected over water and was characterized to find the type of gas produced.

Material used – Plastic bottles (2 Liters capacity, 2 Nos.)

- 5. Slurry collection unit** – This unit was be used to collect the digested slurry of the food wastes at regular intervals for the analysis. This slurry was be analyzed to study there characteristics.

Material required – Plastic beakers (1 Nos.)

Other Materials required – PVC pipes, silicon pipes and valves was be used to connect different units of the plant.

The food wastes was weighed 3 kg (in 2 parts) for the setup. The food waste 3 kg was crushed using a mixer grinder along with diluting it to 7.5 liter with water to form slurry (for each part) (the food waste was diluted at a rate of 1:1.5 with water). The food waste slurry first part was put into thermophilic digester (10L) on the day of starting the digester and second part was stored in fridge and added after 2 days to thermophilic digester, when first part has been passed on to mesophilic digester. 500 ml of diluted cow dung slurry was used as inoculum each time. Total 6 kg food wastes was digested in the setup. The initial analysis of the food wastes before it was put into the reactor were performed, the same day it was collected from the hall. The analysis was done as follows.

- ❖ The diluted food wastes collected on the 1st day were analyzed for total Solids (TS), volatile Solids (VS), total carbohydrates (TC), volatile fatty acids (VFA) and pH.
- ❖ After 2 days of operation in thermophilic setup, the food waste slurry was passed to mesophilic digester, and 250 ml of more inoculum was added to the digester each time.
- ❖ The TS-VS content for each of the digester setup was measured every 5 days from the start of the setup.

Figure 6: Photograph of Portable gas analyzer ACE 9000X-CGA



- ❖ The total carbohydrate content for each of the digester setup was measured using Phenol-Sulfuric acid method every 3 days from the start of the setup.
- ❖ PH was measured every 2 days and was adjusted every 4 days to regulate the pH around 7.
- ❖ The VFA content was measured every 4 days.
- ❖ Gas production of each setup was measured every day.
- ❖ The substrate in the setup was kept well mixed by shaking the setup 4-5 times a day.
- ❖ The gas analyzer used for the analysis of the biogas was portable gas analyzer (Item code: ACE 9000X-CGA).
- ❖ Heater was used for heating the thermophilic digester to required temperature.
- ❖ Vacuum was created in the digesters using vacuum pump to remove air and maintain anaerobic conditions each time after, the tanks pH was adjusted.



Figure 7: Photograph of the Pilot scale setup

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 RESULTS EXPERIMENTAL SET UP (Small Scale)

5.1.1 TS – VS

Table 1: TS – VS change of the small scale setups with time

Day	Setup 1 Mesophilic		Setup 2 Thermo + Meso		Setup 3 Thermophilic	
	TS (% Sample)	VS (% Sample)	TS (% Sample)	VS (% Sample)	TS (% Sample)	VS (% Sample)
0	9.51	9.06	9.51	9.06	9.51	9.06
5	9.04	8.57	7.72	7.3	7.58	7.13
10	8.24	7.66	6.26	5.96	5.84	5.2
15	7.32	6.82	5.19	4.88		
20	6.18	5.64	4.21	3.9		

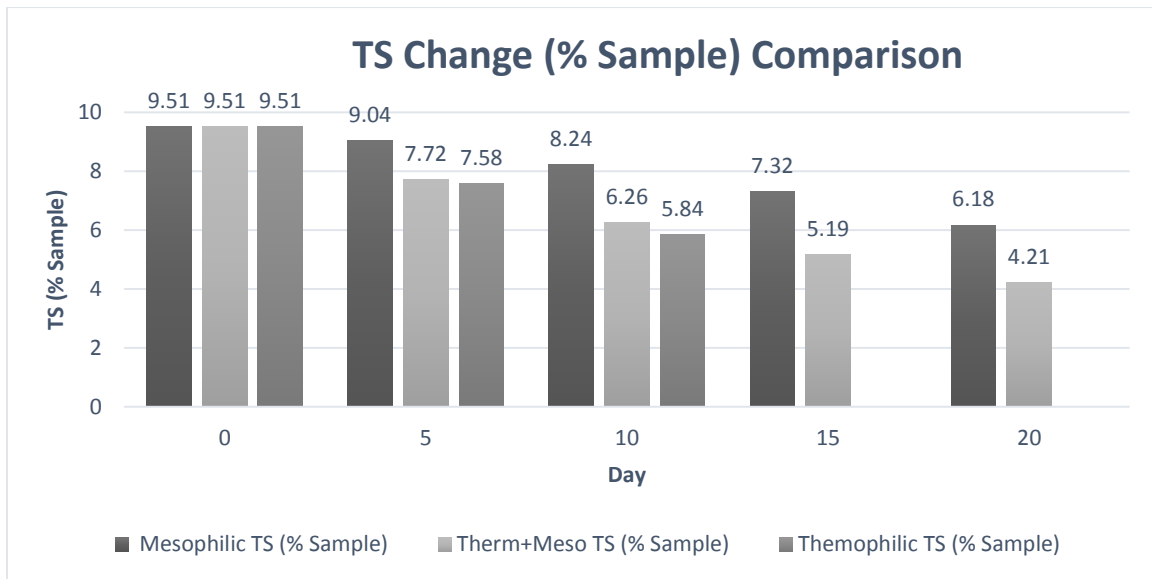


Figure 8: TS Change (% sample) Comparison

Table 1 and figure 8, shows the change in the % change totals solids with respect to sample, for the small scale setups with time. It can be seen that the total solids destruction in case of thermophilic and the two stage setup is more as compared to the mesophilic setup, over whole period of operation.

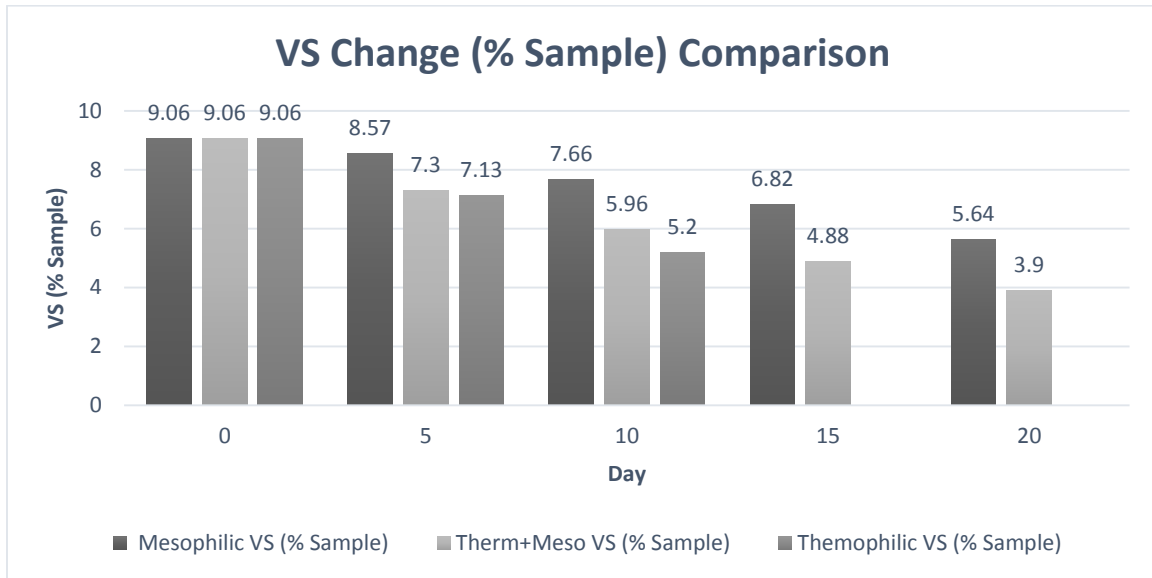


Figure 9: VS Change (% sample) Comparison.

Table 1 and figure 9, shows the change in the % change volatile solid with respect to sample, for the small scale setups with time. It can be seen that the volatile solids destruction in case of thermophilic and the two stage setup is more as compared to the mesophilic setup, over whole period of operation.

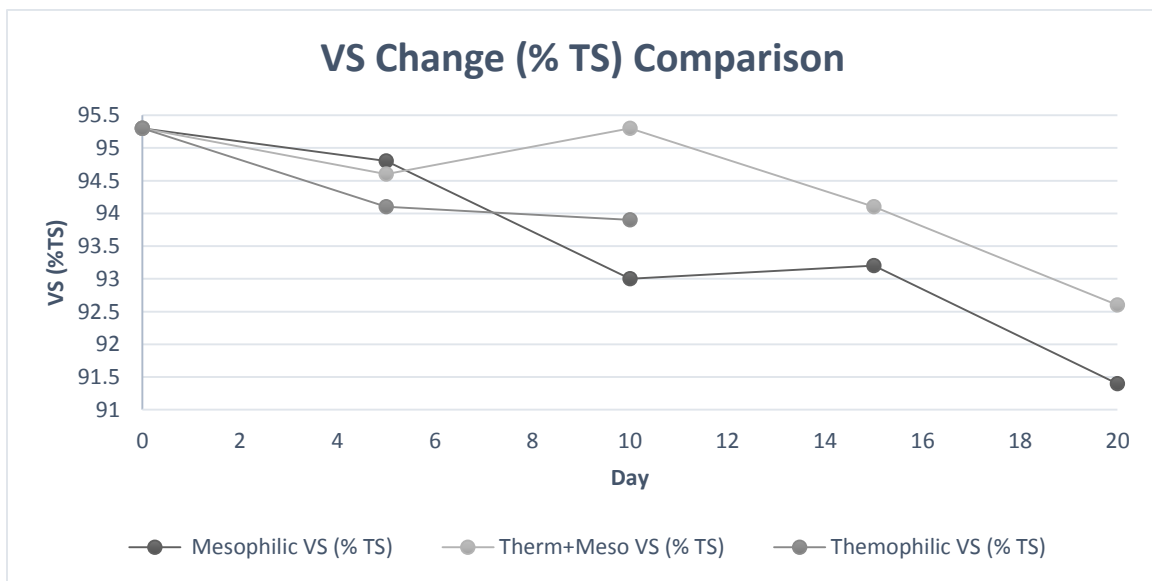


Figure 10: VS Change (% TS) Comparison.

Table 1 and figure 10, shows the change in the % volatile solids with respect to total solids, for the small scale setups with time. It can be seen that the volatile solids destruction in case of the two stage setup faster as compared to the mesophilic setup, over whole period of operation.

5.1.2 pH

Table 2: pH change of the small scale setups with time

Day	pH Setup 1 Mesophilic		pH Setup 2 Therm-Meso		pH Setup 3 Thermophilic	
	Before adjustment	After adjustment	Before adjustment	After adjustment	Before adjustment	After adjustment
0	5.5	6.6	5.5	6.7	5.5	6.5
1	5.2	6.7	4.7	6.5	4.6	6.6
3	5.5	6.9	5	6.8	5	6.6
5	5.7	6.8	5.5	6.8	5.4	6.7
7	5.7	6.9	5.7	7	5.6	6.9
9	5.9	6.8	5.8	6.9	5.9	
11	5.8	7	6.2	7.1		
13	5.8	6.8	6.2	6.8		
15	6.1	6.9	6.5	7		
17	6.2	7	6.3	6.9		
19	6.1	6.9	6.4	7.1		
21	6.3		6.5			

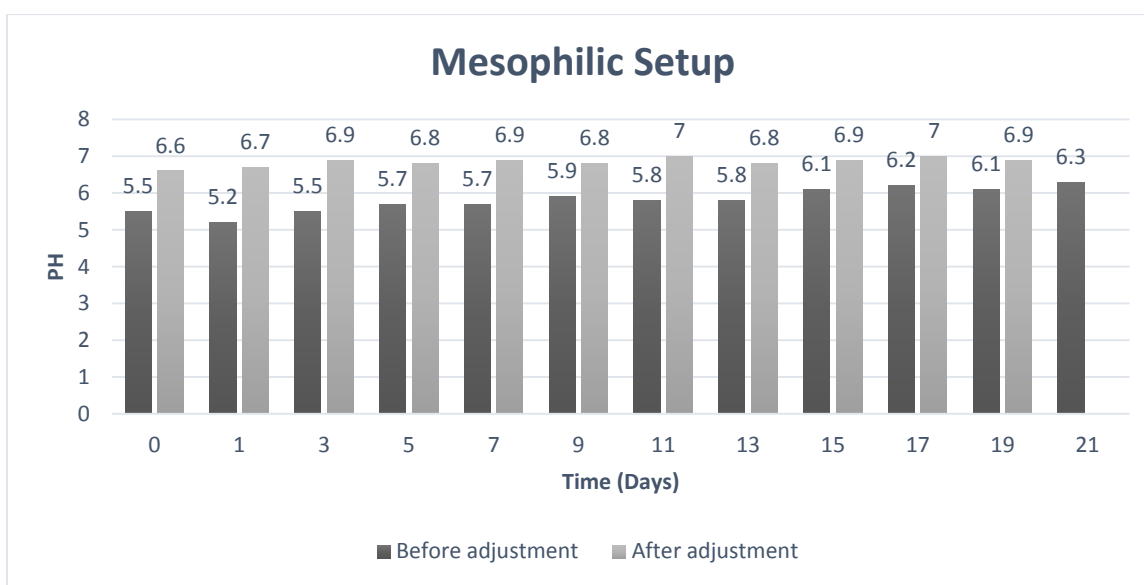


Figure 11: pH change of mesophilic small scale setup with time.

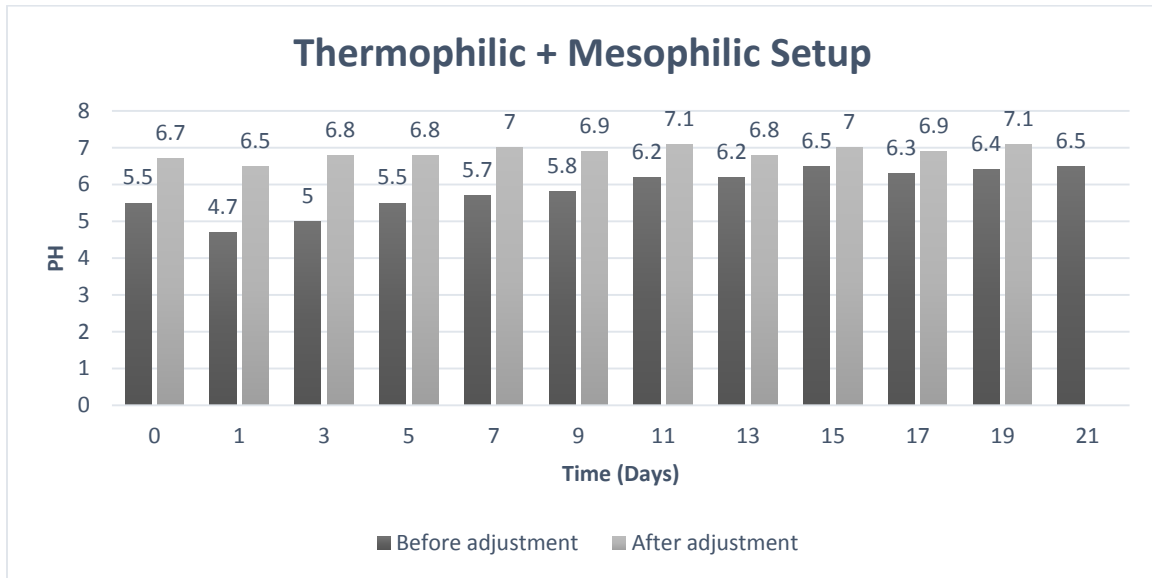


Figure 12: pH change of thermophilic + mesophilic small scale setup with time.

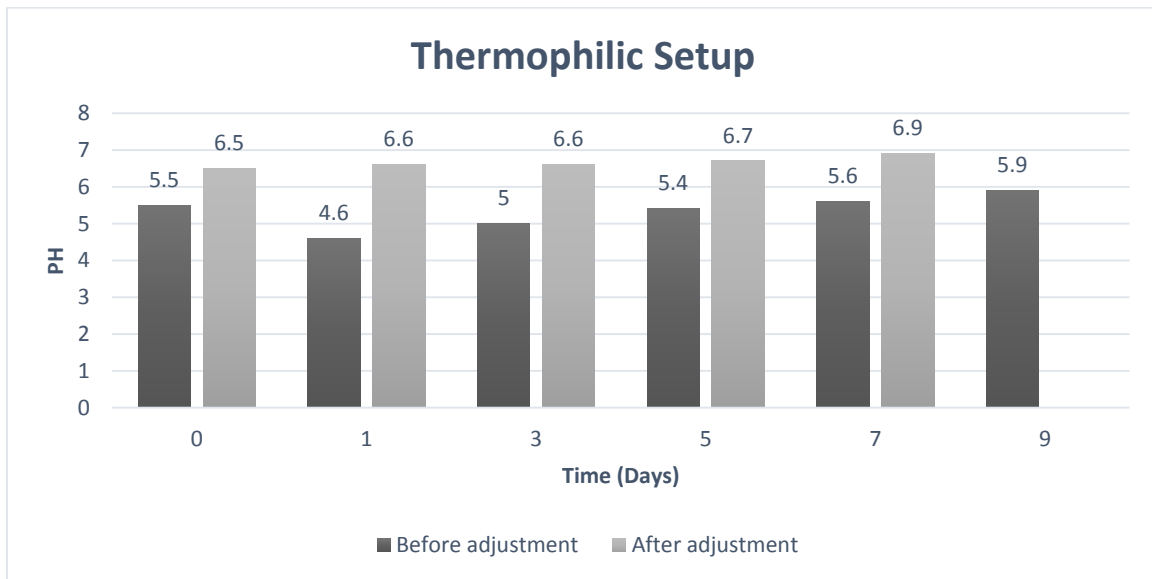


Figure 13: pH change of thermophilic small scale setup with time

Figure 11-13, show the change of pH for the three setups with respect to time over the operational period. The pH can be seen to decrease fast even after adjustment. This signifies the production of large amount of acids initially The pH drop starts to stabilize after 10 days for two stage setup and after 15 days for mesophilic setup.

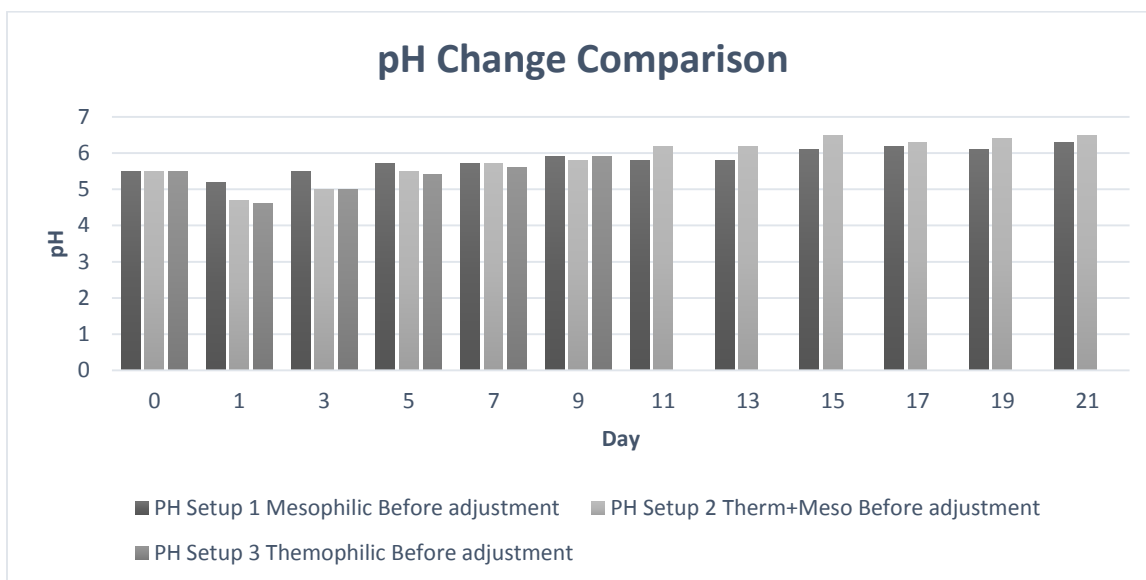


Figure 14: pH change of comparison of small scale setup with time

Table 2 and figure 14, compares the change in pH of the three setups with respect to time. The pH can be seen to be lower for the two stage setup initially but can be seen to stabilize faster than the mesophilic setup.

5.1.3 Carbohydrates Change

Table 3: Carbohydrates change of the small scale setups with time

Day	Setup 1 Mesophilic (g/l)	Setup 2 Therm+Meso (g/l)	Setup 3 Thermophilic (g/l)
0	56.6	56.6	56.6
3	53.4	49.2	50.1
6	49.3	46.1	45.8
9	46.1	41.7	39.3
12	42.8	36.4	
15	37.7	31.9	
18	31.2	24.6	
21	26.5	17.9	

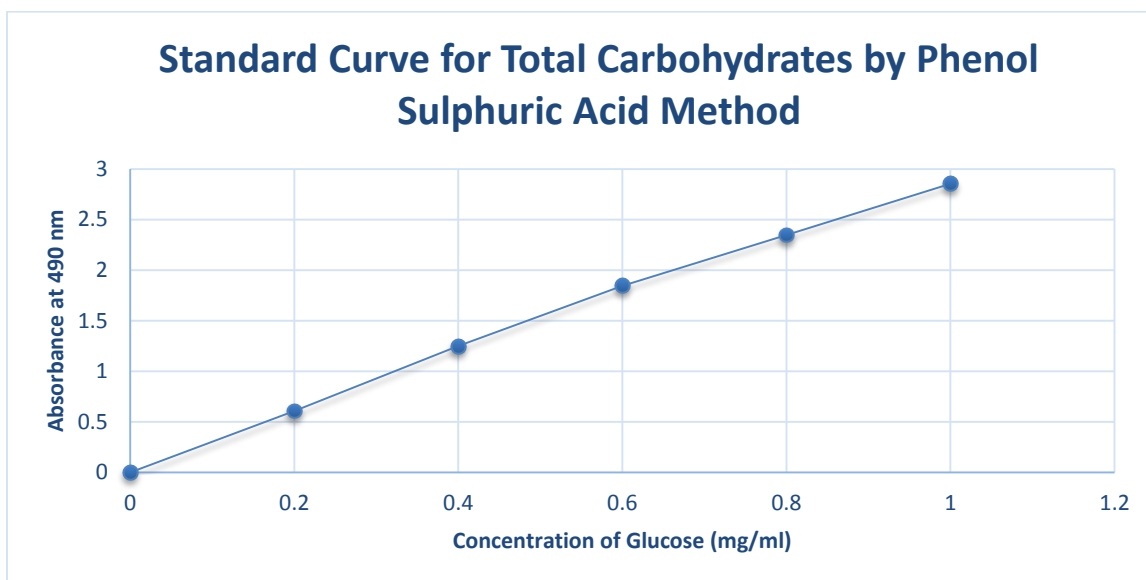


Figure 15: Standard curve for total carbohydrates by phenol sulfuric acid method

Figure 15 shows the Standard curve of glucose from which the carbohydrates content has been measured.

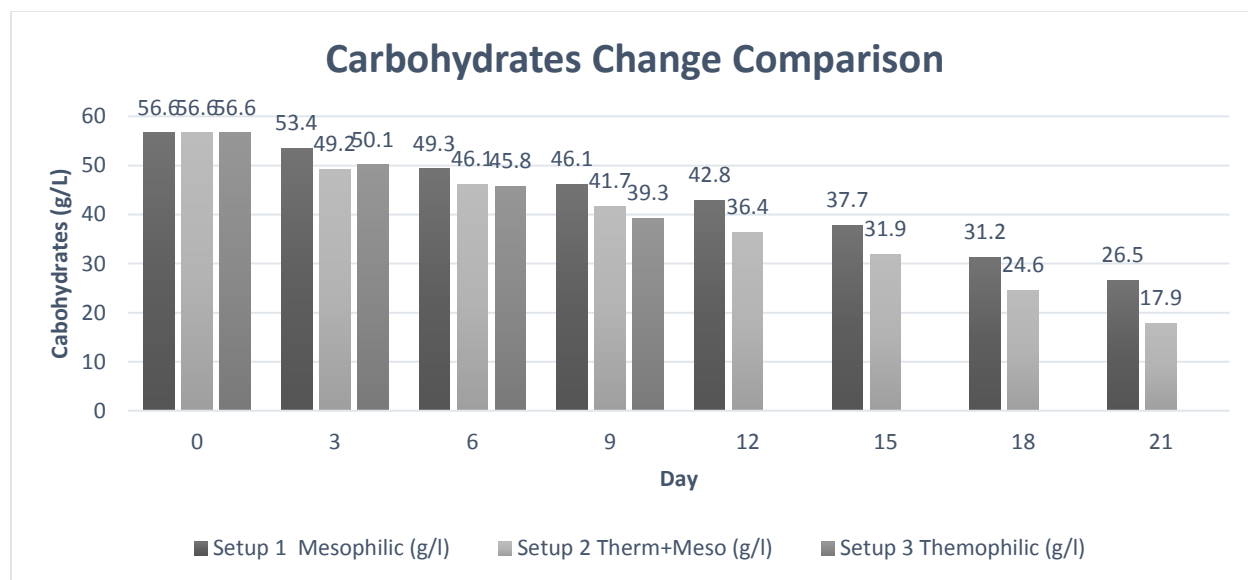


Figure 16: Carbohydrates change comparison of small scale setups with time

Table 3 and figure 16, compares the change in carbohydrate content of the three setups with respect to time. It can be seen that the carbohydrates content in case of thermophilic and the two stage setup is reducing faster as compared to the mesophilic setup, over whole period of operation. This signifies faster decomposition in case of the latter setups.

5.1.4 Gas Production

Table 4: Gas Production of the small scale setups with time

Day	Setup 1 Mesophilic (ml)	Setup 2 Therm+Meso (ml)	Setup 3 Thermophilic (ml)
1	90	600	540
2	100	550	500
3	120	400	480
4	200	400	470
5	230	390	430
6	230	410	430
7	280	380	420
8	300	400	430
9	290	420	400
10	300	410	400
11	320	410	
12	310	460	
13	320	450	
14	330	450	
15	350	470	
16	330	460	
17	350	450	
18	370	430	
19	360	430	
20	360	420	

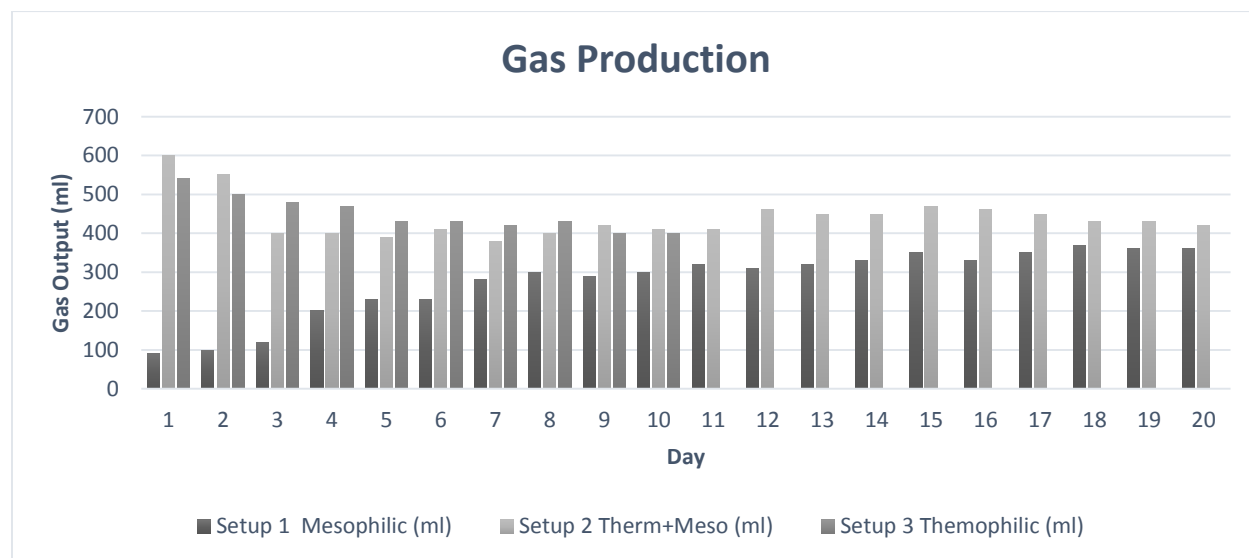


Figure 17: Gas Production comparison of the small scale setup with time

Table 4 and figure 17, compares the gas production from the three setups with respect to time. It can be seen that the gas produced is higher initially in case of thermophilic and the setup no. 2. Then the gas production drops significantly when the setup is shifted from thermophilic to mesophilic stage. The gas production in case of the mesophilic setup starts slowly and increases gradually and stabilizes after 15 days.

5.2 RESULTS EXPERIMENTAL SET UP (Pilot Scale)

5.2.1 TS – VS

Table 5: TS – VS change of the pilot scale setup with time

Day	TS (% Sample)	VS (% TS)	VS (% Sample)
0	10.27	94	9.65
5	8.72	93.1	8.11
10	7.26	92.3	6.7
15	6.19	93	5.76
20	5.78	92.6	5.35
25	5.51	92.1	5.07

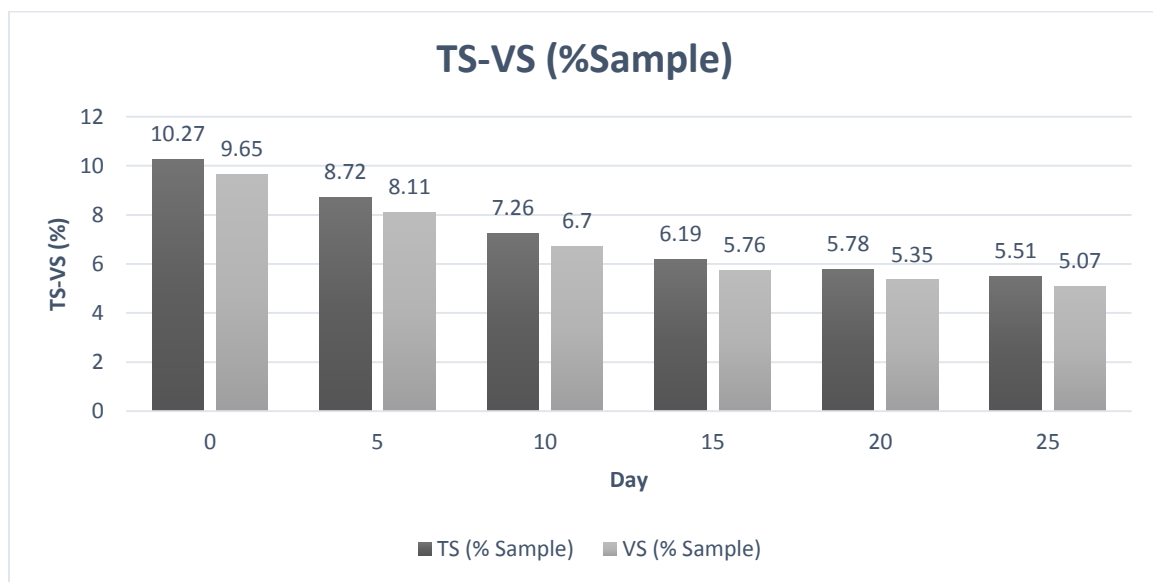


Figure 18: TS – VS (%Sample) change of the pilot scale setup with time.

Table 5 and figure 18, shows the change in the total solids with respect to time & volatile solids % change with respect to sample, for the pilot scale setup. It was observed that the TS-VS destruction is faster initially but slows down with time over the operation period. Initially TS was 10.27 % of sample and VS was 9.65 %. The TS and VS destruction was found to be almost 50% of the initial value on 25th day of the operation of reactor.

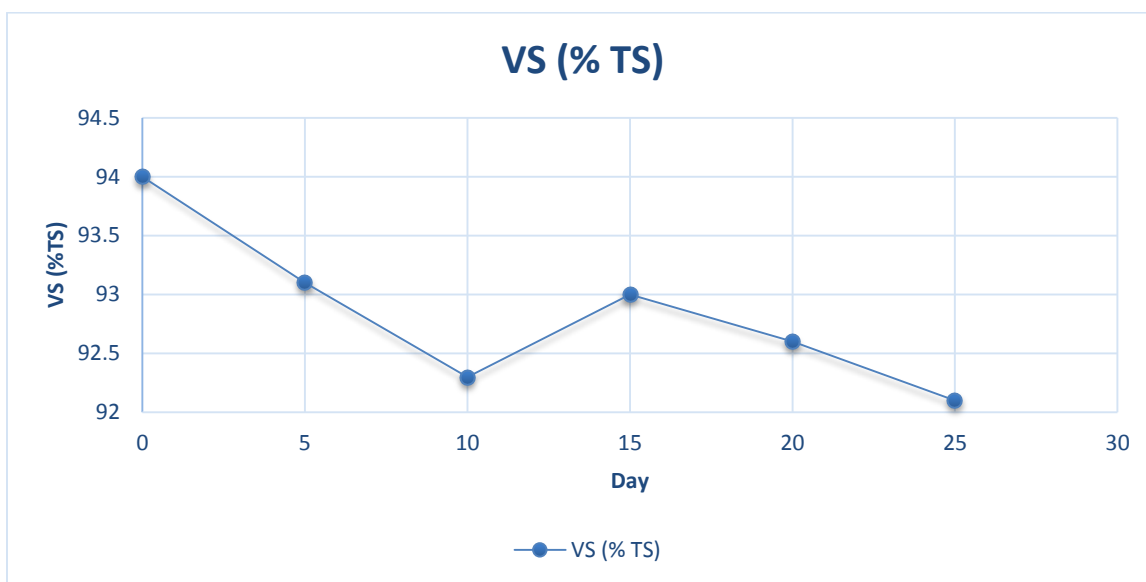


Figure 19: VS (%TS) change of the pilot scale setup with time.

Figure 19 shows the % change in the Volatile solids with respect to total solids, for the pilot scale. It was observed that the volatile solids content changed from 94% to 92.1%.

5.2.2 pH

Table 6: pH change of the pilot scale setup with time

Day	PH		Day	PH	
	Before adjustment	After adjustment		Before adjustment	After adjustment
0	5.4	6.7	13	6.2	
1	5.2		15	6.1	6.8
3	4.6	6.7	17	6.3	
5	5.8		19	6	7.1
7	5.3	6.8	21	6.5	
9	5.8		24	6.6	
11	5.9	6.9			

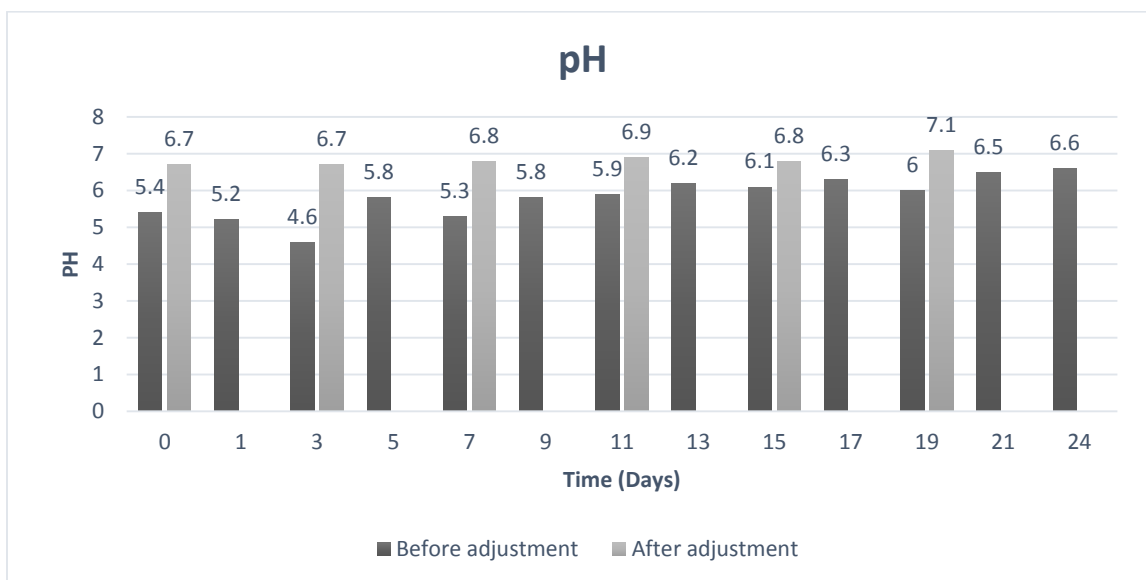


Figure 20: pH change of the pilot scale setup with time.

Table 6 and figure 20, shows the change in pH of the setup with respect to time, for the pilot scale setup. The pH can be seen to drop below 5 initially signifying large amount of acid production. The pH even after adjustment drops significantly but stabilizes above 6.0 pH after 15 days of operation of the setup.

5.2.3 Carbohydrates

Table 7: Carbohydrate change of the pilot scale setup with time

Day	Carbohydrates (g/L)	Day	Carbohydrates (g/L)
0	61.2	15	35.9
3	54.7	18	30.2
6	51.3	21	26.1
9	49.7	24	22.3
12	41.4		

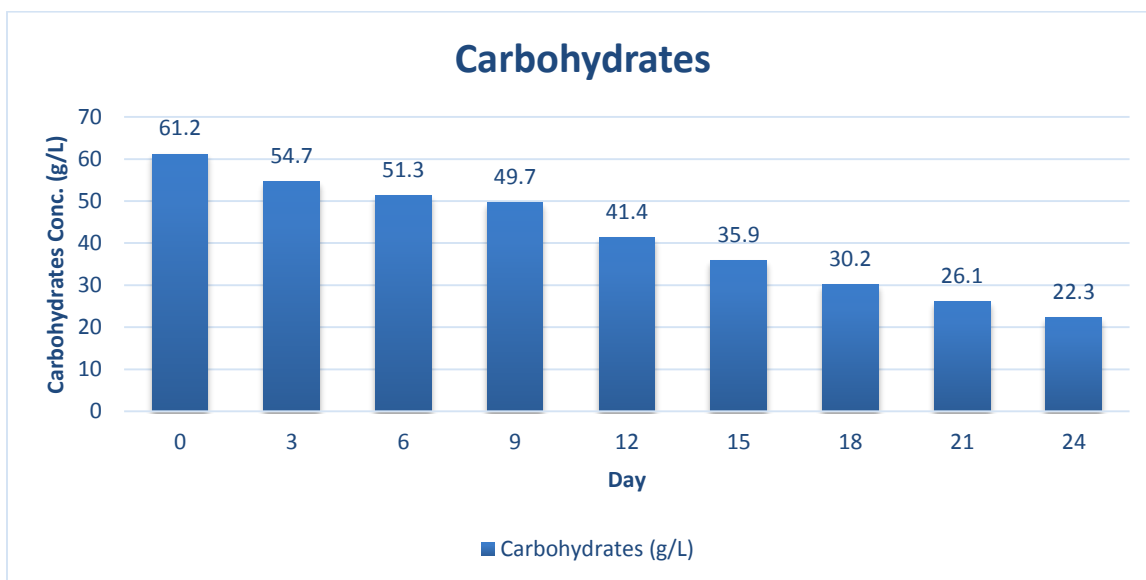


Figure 21: Carbohydrates change of the pilot scale setup with time

Table 7 and figure 21, shows the change of carbohydrate content of the setup with respect to time, for the pilot scale. The carbohydrate content can be seen to decrease rapidly initially but slows down eventually. The carbohydrates decreased from 61.2 g/L to 22.3 g/L on 25th day of reactor operation. Rapid decrease signifies faster degradation of carbohydrates.

5.2.4 Gas Production

Table 8: Gas Production of the pilot scale setup with time

Day	Gas Production	Day	Gas Production
1	7300	14	7050
2	7150	15	7450
3	4950	16	7100
4	5100	17	7200
5	4750	18	7400
6	5450	19	7350
7	5850	20	7450
8	6150	21	7300
9	6200	22	7250
10	6350	23	7350
11	6100	24	7150
12	6750	25	7400
13	7100		

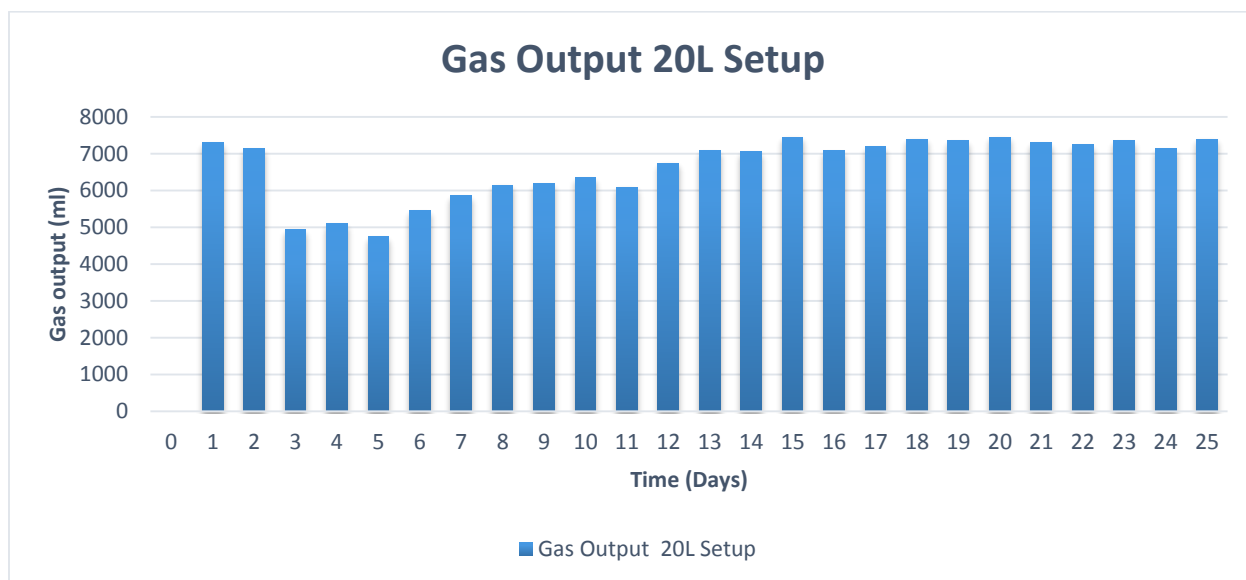


Figure 22: Gas Production of the pilot scale setup with time

Table 8 and figure 22, shows the gas production from the setup, with respect to time, for the pilot scale. It can be seen that the gas produced is higher initially in case of thermophilic stage. Then the gas production drops significantly when the setup is shifted from thermophilic to mesophilic stage. The gas production increases gradually and stabilizes after 15 days. Maximum gas produces during the operation of the setup was ~7.45 L.

5.2.5 VFA

Table 9: VFA change of the pilot scale setup with time

Day	VFA (mg/L)
0	2475.5
4	3873.5
8	4421.5
12	4565
16	4758.5
20	4769.5
24	4954

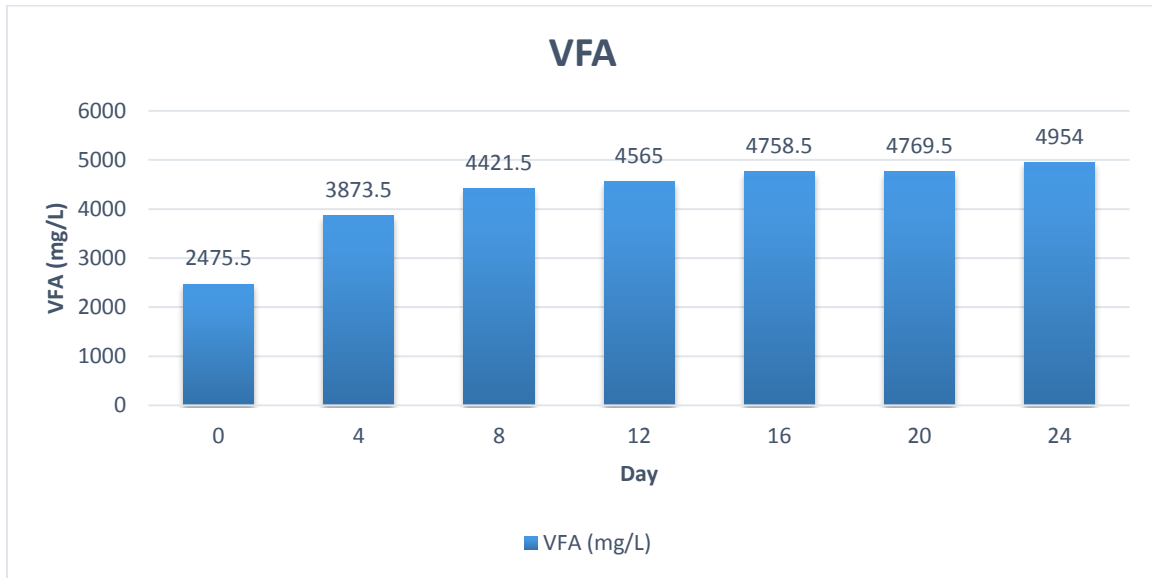


Figure 23: VFA change of the pilot scale setup with time

Table 9 and figure 23, shows the VFA change of the setup with respect to time, for the pilot scale. VFA content can be seen to increase consistently. This signifies the accumulation of the VFA in the setup.

5.2.6 Gas Analysis

Table 10: Gas analysis of the pilot scale setup with time

Day	H ₂ (%)	CO ₂ (%)	CH ₄ (%)	CO (%)	O ₂ (%)
5	3.12	66.21	0	0.01	6.21
12	0	70.13	1.1	0.01	7.95
18	0	55.87	10.19	0.01	8.17
25	0	45.29	18.66	0.01	6.36

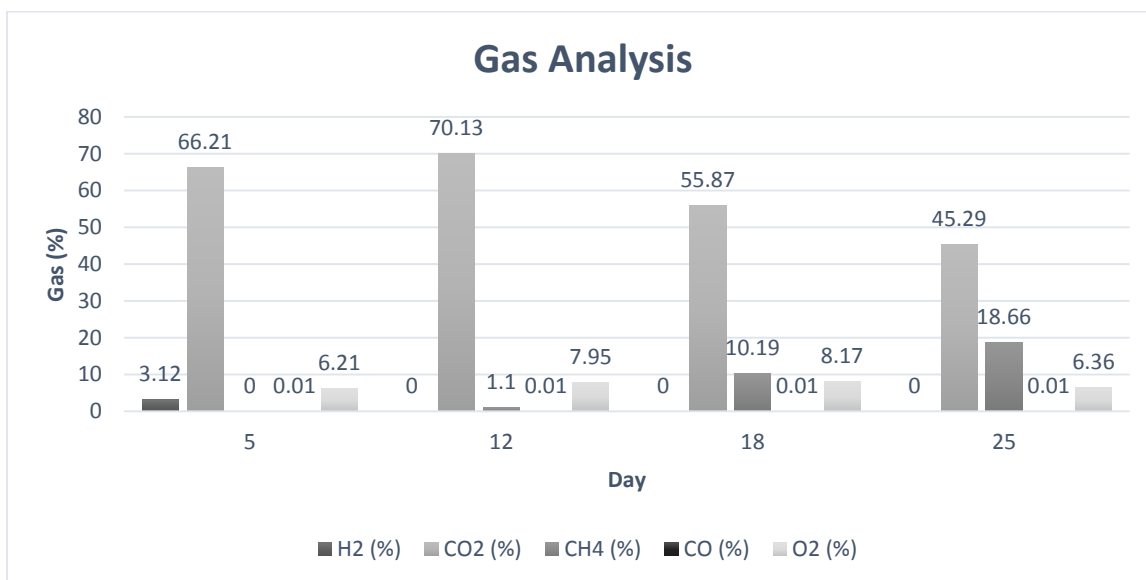


Figure 24: Gas analysis of the pilot scale setup with time

Table 10 and figure 24, shows the gas analysis of the setup with respect to time, for the pilot scale. Presence of hydrogen has been observed initially. The carbon dioxide content was maximum over the operation period. The methane concentration in biogas has been found to be low, might be due to accumulation of VFA. The concentration of methane may not have been correctly measured due to low sensitivity of the instrument used for the analysis. The presence of carbon monoxide signifies the impurities in the gas produced, or it may have come from the air contamination.

CHAPTER 6

CASE STUDY

6.1 SURVEY OF HOSTEL MESSES

The survey of the messes of the hostels of NIT was done to determine the amount of food waste generation from the hostels. The food waste generation and the number of cylinders of LPG used at these 5 messes was estimated on daily basis mentioned below during a week in month of March 2014.

The uncooked wastes mainly consisted of vegetable remains after cutting, onion peels, etc.

The cooked wastes mainly consisted of leftover rice, vegetables, pulses, roti, bread, etc.

Table 11: Waste generation and LPG usage at the hostel messes.

Day	Hall 8 (VS)			Hall 7 (HB)			Hall 4/5 (DBA/MSS)		
	Uncooked Wastes (L)	Cooked Wastes (L)	LPG Cylinder (14.2 L)	Uncooked Wastes(L)	Cooked Wastes (L)	LPG Cylinder (14.2 L)	Uncooked Wastes (L)	Cooked Wastes (L)	LPG Cylinder (14.2 L)
1	150	350	12	150	150	6	150	150	9
2	150	300	11	150	150	5	150	150	8
3	150	300	12	150	150	6	100	200	9
4	200	350	12	150	150	6	100	150	8
5	150	300	11	150	150	5	150	200	8
6	150	350	12	150	150	6	150	150	9
7	200	400	14	200	200	7	200	250	10
Total	1150	2350	84	1100	1100	41	1000	1250	61
Average/ day	164	336	12	157	157	6	143	179	9
Day	Hall 2/3 (MV/GDB)			Hall 6 (CVR)					
	Uncooked Wastes(L)	Cooked Wastes (L)	LPG Cylinder (14.2 L)	Uncooked Wastes(L)	Cooked Wastes(L)	LPG Cylinder (14.2 L)	HALL Mess Name		No. of Students
1	150	300	9	200	250	8	Hall 8 (VS)		1300
2	200	250	8	150	300	9	Hall 7 (HB)		550
3	150	300	8	150	250	9	Hall 4/5 (DBA/MSS)		850
4	150	250	9	200	300	8	Hall 2/3 (MV/GDB)		950
5	200	300	8	150	300	8	Hall 6 (CVR)		1000
6	150	300	9	150	300	9			
7	200	350	10	200	350	10			
Total	1200	2050	61	1200	2050	61			
Average/ day	171	293	9	171	293	9			

Note: The above data is just an estimate and has not been measured by using any instruments. The waste generation was estimated on the basis of number of containers of waste collected every day.

The amount of waste generation depends on several factors:

- The type of food items made on that particular day.
- The food waste generation varies at various times of the year depending on the number of students the messes are serving. (For example: the food waste generated during summer season will be very low because number of students present is very less.

From the above data it can be estimated that:

Total cooked wastes generated from 5 messes serving 4650 students/day = ~1300L/day

Total uncooked wastes generated from 5 messes serving 4650 students/day = ~800L/day

The total amount of LPG gas used per day = (14.2L/cylinder * 45) = ~640L/day

6.2 ANALYSIS

From our experimental pilot scale setup it can be concluded gas production of ~7.5 liters/day can be produced from a ~6L (6kg) of food wastes per day under stable and ideal conditions (like maintaining pH, temperature, etc.).

Considering that uncooked wastes generated are partly not suitable for digestion in biogas plants and they have low density implying lower weight. The cooked wastes generated contain a lot of water almost 75%. If only half of the uncooked wastes and the total cooked wastes are usable for the biogas plant, let us assume that we have total of 1500L of wastes digestible in the digesters.

Considering and assuming the above situations and ideal conditions of digester operation we can estimate:

Total biogas production from about 1500L of food wastes = ~1800L of biogas per day can be obtained by setting up a large scale biogas plant at our institute. Assuming the following values:

“Calorific value of Biogas = 6 kWh/m³”

“Calorific value of LPG = 26.1 kWh/m³”

Total energy produced from 1800 L biogas per day = 10.8 kWh/m³

The energy obtained from biogas is equivalent to ~0.6 m³ LPG.

Result: From the above data we can estimate that we can save almost ~27 cylinders of LPG every day if we use the total food waste generated at our hotels to produce biogas under ideal operational conditions.

CHAPTER 7

CONCLUSION

The Biogas setup based on kitchen wastes was implemented on small scale setups to find the effects of the process parameters on the biogas production. It was found that the pH and temperature conditions had huge influence on the working of the biogas plant. In 1st experimental setup a comparison of the digestion of food wastes in small scale was done. In this experiment three setups were compared under mesophilic, thermophilic and two stage mesophilic and thermophilic conditions. The kitchen waste digestion in case of two stage thermophilic and mesophilic setup was found to be 30% faster than mesophilic setup. This difference was observed with respect to total solids and carbohydrates change over the operation period. The gas production initially was found to be 40% faster in case of two stage setup than the mesophilic setup.

The pilot scale plant was based on two stage thermophilic and mesophilic digestion process and operated as a batch reactor under controlled conditions of pH and temperature. A constant rate of gas production was achieved but accumulation of volatile fatty acids was also observed. In batch condition, it was observed that a maximum of 7.45 Liters biogas was produced from the digestion of 6 kg of food wastes in 25 days. Initially the total solids of the waste slurry was measured to be 10.27% on the day of starting the reactor and 5.51% on 25th day. The total carbohydrates degradation was measured to be 61.2 g/L on the day of starting the reactor and 22.3 g/L on 24th day. The volatile fatty acid concentration was measured as 2475.5 mg/L on the day of starting the reactor and 4954 mg/L on 24th day.

The gas analysis results were not satisfactory. It was expected that in batch conditions the digestion of one batch of feed substrate should have completed within 15 days. It was observed from the analysis of various characteristics that the digestion in the reactor was much slower than expected. The gas analysis data showed only 18.66 % methane gas concentration on 25th day. The possible reason for the low amount of methane gas formation may be due to accumulation of volatile fatty acids. Hence it is suggested the reactor should be operated for more than 3 months in continuous mode to obtain better results.

The future prospect of the project can be the improvement of biogas production from kitchen wastes by incorporating additives and optimization of other process parameters in the two stage anaerobic digestion process.

The survey of the NIT hostels for the data of the food wastes generated showed large amount of food wastes produced at our hostels that can be treated by setting up a large scale two stage biogas plant, for production of biogas, taking into view the successful implementation of such biogas plant at Bhabha Atomic Research Center (BARC) premises.

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